

# Filament-Winding Plastics

## Part 4 - Syntheses, Properties, and Uses of m-Aminobenzyl Amine (MABA)

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"Part 3 — Some Effects of Curing Agent Structure Upon Epoxide Plastic Physical Properties," J. R. Griffith, NRL Report 6313, Sept. 1965

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**Abstract:** During research at NRL aimed at improving the tensile properties of plastics suitable for filament winding applications, about 100 materials have been examined in various combinations. The majority of the materials offered no outstanding improvements in the properties sought, relative to standard filament winding systems. But one material new to this application, *m*-aminobenzyl amine or MABA, has been found to impart substantial improvements to two epoxy plastics of the most useful types.

MABA may be synthesized readily by catalytic hydrogenation of *m*-nitrobenzonitrile or *m*-nitrobenzaldoxime. It is a solid which melts below 40°C and may be easily incorporated into epoxy resins. Its unique properties apparently result from the differences in reactivity of the two amino groups, one attached to the benzene ring and the other displaced by a methylene group. MABA and the diglycidyl ether of Bisphenol-A produce a liquid resin system suitable for "wet winding," from which plastic of exceptional energy-absorbing capability is obtained. The plastic produced from MABA and an isomer of dicyclopentyl ether diepoxide has an exceptionally high tensile modulus and ultimate tensile strength. This system has convenient properties for the production of preimpregnated glass strands from which filament windings can be fabricated.

## INTRODUCTION

At NRL two direct syntheses of *m*-aminobenzyl amine, by hydrogenation of readily produced intermediates, have been developed to the stage of small pilot plant production. As a new curing agent for epoxy resins, this compound has produced plastics which exhibited extraordinary properties including (a) exceptional elongation at high tensile loads, resulting in enhanced resistance to crack propagation (1), (b) very high tensile modulus and ultimate tensile strength, and (c) curing temperatures lower than those required for commonly used aromatic amines, resulting in reduced internal strains in glass fiber composites. At present, there are no known commercial sources for *m*-aminobenzyl amine.

During the course of research on epoxy plastics for filament winding applications, approximately 100 materials have been investigated, including curing agents, resins, flexibilizers, and diluents.

The particular objective of the work has been that of finding usable systems which would display tensile properties in excess of those obtained with conventional systems. *m*-Aminobenzyl amine imparts exceptional elongation at a high tensile level to the plastic produced with the diglycidyl ether of Bisphenol-A (2). Because the plastic exhibits a yield point at the 12,000-psi tensile level, its total energy absorption at break is more than 50% greater than that of the analogous plastic produced with *m*-phenylene diamine. With one isomer of dicyclopentyl ether diepoxide, the *m*-aminobenzyl amine produces a plastic with a tensile strength of 20,100 psi, a tensile modulus of 766,000 psi, and an elongation of 5% (3). The corresponding values for the similar plastic produced with *m*-phenylene diamine are 17,600 psi, 684,000 psi, and 4.6%, respectively. This represents an unusual case in which tensile strength and modulus are both increased without a corresponding loss in elongation. Since published data have not been encountered regarding plastics suitable for filament winding applications with strength properties in excess of those cited above, a summary evaluation of *m*-aminobenzyl amine is presented.

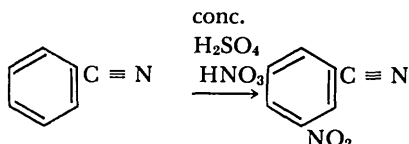
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NRL Problem C03-14; Project RR 007-08-44-5508. This is an interim report; work is continuing on the problem. Manuscript submitted May 18, 1966.

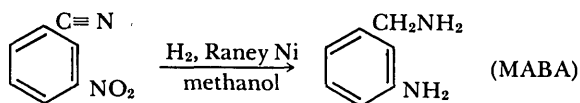
## SYNTHESES OF MABA

### *m*-Nitrobenzonitrile Method

Benzonitrile was nitrated in the meta position with mixed acids by the method of Bogert and Beans (4):



Hydrogenation of both pendant groups occurs readily in the presence of Raney nickel prepared by the method of Mazingo (5). Ethyl acetate or, preferably, methanol may be used as solvent:

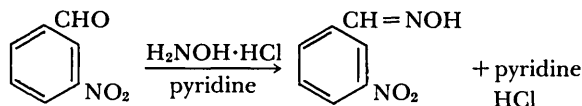


In a typical experiment, 150 g (1 mole) of *m*-nitrobenzonitrile, 700 ml of methanol, and 7 g of Raney nickel slurry in ethanol were placed into a hydrogenation bomb equipped with a heater, stirrer, and cooling coils. The bomb was thoroughly outgassed with hydrogen and a pressure of 400 psig was established. After 10 minutes' stirring, the temperature of the reaction contents began to rise steadily. The hydrogen pressure was maintained at 400 to 500 psig and the temperature was controlled between 80 and 100°C, by heating or cooling as necessary, for 2 hours. After this time, hydrogen consumption became very slow, and the reaction was discontinued. The catalyst was removed from the dark-brown reaction solution on a porous glass suction funnel, the solvent was removed by a rotary evaporator, and the residue was a dark oil. Vacuum distillation of this residue through a short column filled with glass helices gave a major fraction at 133-135°C (5 mm). The product, 60 g (49%), was a clear, slightly green oil, which crystallized upon being cooled. Redistillation of a small quantity yielded a product which melted at 37-38°C. The dibenzoyl derivative was prepared with alkaline benzoyl chloride, and it melted at 173-175°C (reported (6) m.p., 173-174°C). The yield is affected somewhat by the reduction conditions, supposedly because of the formation of secondary amines from con-

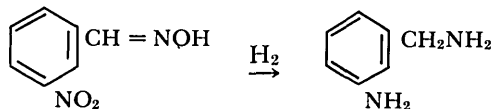
densation of initial product with the nitrile group. In all cases, there remained a very viscous, dark-brown residue in the distillation flask. In a few preparations of MABA by this method, 60% yields were obtained, but this did not occur consistently. A recommended fast reaction at higher pressures and temperatures for nitrile reduction (7) did not improve the yield.

### *m*-Nitrobenzaldehyde Method

In an alternate procedure, *m*-nitrobenzaldehyde was reduced to MABA. The starting compound for this synthesis was *m*-nitrobenzaldehyde, a commercially available material. On a small scale, this oxime is more easily prepared than the *m*-nitrobenzonitrile in the preceding method:



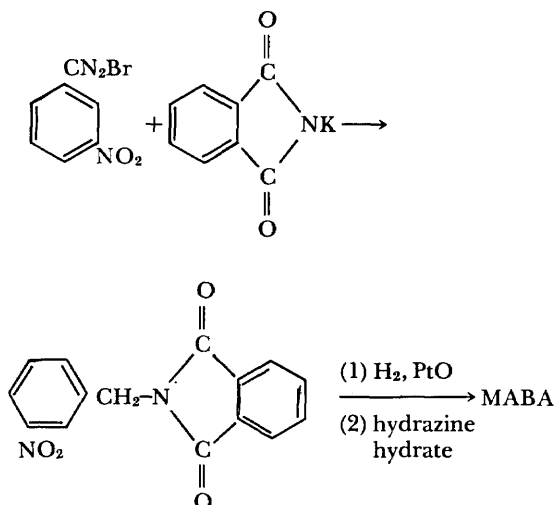
In a typical preparation, 200 g (1.33 mole) of *m*-nitrobenzaldehyde, 115 g of hydroxylamine hydrochloride, 150 ml of pyridine, and 350 ml of ethanol were placed into a 2000-ml flask fitted with a stirrer and a reflux condenser. The flask contents were stirred and refluxed for 3 hr. Ethanol was then removed on a rotary evaporator, and the resulting crystalline residue was washed on a suction funnel with water until the wash solution was colorless. The yield of oxime was 214 g (97%). This material was then hydrogenated in methanol at 400-500 psig and 80-100°C for 6 hr, over Raney nickel:



Separation and purification of the product as in the preceding method yielded 82 g (52%) of MABA.

### Gabriel Method

Kornblum and Iffland (6) prepared MABA from *m*-nitrobenzyl bromide by the Gabriel phthalimide synthesis:



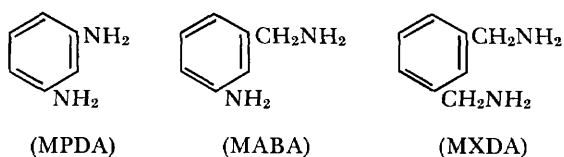
This procedure is more involved than the direct hydrogenations in the two preceding methods, and the overall yield was 28%.

### PHYSICAL PROPERTIES

Freshly prepared MABA is a white, crystalline solid with a somewhat waxy appearance and a melting point under 40°C. The melt is clear, with the appearance of water. At room temperature it does not fume when exposed to the atmosphere, but it has a characteristic amine odor. It dissolves readily in water, ethanol, and chloroform, dissolves slowly in benzene and ethyl ether, and is insoluble in carbon tetrachloride. The density at 25°C is 0.997, and the refractive index is 1.6048. A slowly cooled melt will supercool and remain liquid at room temperature if neither seeded nor agitated. Equal weights of MABA and *m*-phenylene diamine form a stable, fluid solution at temperatures above 15°C.

### CHEMICAL PROPERTIES

Most of the chemical properties of MABA are intermediate to those of its homologs, meta-phenylene diamine (MPDA) and metaxylylene diamine (MXDA):



MPDA is essentially an aromatic amine, while MXDA has properties which are typical of aliphatic amines. For instance, MXDA is a liquid which readily absorbs carbon dioxide from the atmosphere to produce insoluble carbonates, whereas MPDA does not. MABA will also extract carbon dioxide from the air, but either the rate of extraction is much slower than that of MXDA or the resulting carbonate is more soluble in the liquid MABA. A practical consequence of this is that castings of epoxy resins cured with MXDA in the presence of air are milky, while those produced with MPDA and MABA are transparent. On the other hand, MPDA discolors readily, whereas MXDA does not. Thus, castings made with MPDA are usually brown, while those made with MXDA are nearly colorless. Freshly made MABA produces castings only slightly darker than those of MXDA. A major disadvantage of MABA, however, is that it discolors over a period of time at room temperature more readily than MPDA or MXDA. However, it can be stored in airtight containers under refrigeration indefinitely. Under the conditions employed to form a "B"-stage resin with the diglycidyl ether of Bisphenol-A, MABA discolors slightly, and during cure at higher temperatures the color deepens, but much less than that of a similar MPDA system.

Reaction rates and exotherms with glycidyl epoxies are also intermediate for MABA between those of MPDA and MXDA. A sizable mass of liquid containing MPDA reacts slowly to achieve "B" stage, whereas MXDA is somewhat treacherous in that a sudden, hot reaction may occur. MABA is similar to MXDA, since the reaction begins immediately upon mixing of the resin components; however, the "runaway" reaction can be prevented by temperature control. When a cylindrical sample of stoichiometric MABA and the diglycidyl ether of Bisphenol-A, 5 cm in diameter and 5 cm long, was prepared at 25°C, its temperature rose steadily at a rate of 1/4°C per minute for an hour, and then increased to an average rate of 3/4°C per minute for an additional hour. The peak temperature, 84°C, occurred 2 hours after mixing, and gelation occurred at that temperature. In winding operations, 400-g samples have been cooled with copper coils through which water at 25°C was passed. In spite of the continuous agitation of the resin due to

the passage of glass strands, an uncontrolled solidification has not occurred.

Because of the aliphatic amine reaction characteristics of MABA, optimum cure of the plastic produced with the diglycidyl ether of Bisphenol A may be obtained in 3 hours at 120°C. Optimum cure with MPDA requires about 3 hours at 150°C. Since the level of internal stress within a composite structure depends on the maximum temperature required to cure the plastic (8), composites produced with MABA are in a lower state of stress at room temperature than those produced with MPDA.

A stoichiometric blend of MABA with the liquid isomer of dicyclopentyl ether diepoxide reacts slowly over a period of several weeks at room temperature. Because of this characteristic, the major problem involved in the use of this type epoxy, *i.e.*, excessive volatility at elevated temperature, can be circumvented. If a freshly made sample of the liquid resin is heated at 160°C for 1 hour, solidification occurs, but the sample loses about 30% of its total weight because of volatilization of the epoxy. However, if the sample is allowed to thicken for 3 weeks and is then placed into an oven at 160°C, it cures with the loss of only 3 or 4% of its weight. At the end of the storage time at ambient temperature, the system is a soft, amorphous prepolymer which may be melted or dissolved in solvents. "Prepreg" may be prepared by dissolving it in solvent and passing glass through the solution. After the solvent is evaporated, a flexible, tacky strand is obtained which is suitable for winding. Such a treated strand or roving may be retained in a flexible condition indefinitely by refrigeration. At ambient temperature it will eventually become brittle and unsuitable for use.

A similar process may be used to prepare prepolymers and impregnated glass fibers with MABA and 1,4-cyclohexadiene diepoxide, but the conditions required are more critical. This epoxy is a crystalline solid at room temperature which sublimates readily upon being heated and precipitates readily from a catalyzed blend at room temperature. Consequently, in the formation of the prepolymer, a closed container must be maintained at the minimum temperature required to keep the system homogeneous until sufficient reaction has occurred to prevent phase separation when the temperature is lowered. Thus a stoi-

chiometric solution of MABA and the epoxy was maintained at 72°C for 5 hours to yield a viscous liquid which became a transparent, light-yellow glass upon being cooled to 25°C. This prepolymer could be cured at 150°C for 3 hours with a weight loss of only 1 or 2% from an open container. It was soluble in ethanol, and this solution was used to prepare preimpregnated glass fibers. After the solvent was removed, however, the treated fibers were stiff at room temperature. A processing temperature of about 70-75°C was required to keep the system flexible enough for use in winding.

#### **DESIRABLE PROPERTIES OF CURING AGENTS FOR FILAMENT WINDING RESINS**

Curing agents for epoxy resins must meet a few critical requirements to be useful in filament-winding applications. A prospective curing agent must have a proper combination of chemical and physical properties before it can be incorporated into a plastic under realistic conditions. If it is a solid at ordinary temperatures, some means of obtaining a molecularly homogeneous mixture with an epoxy resin must be available. Whether liquid or solid, it must be soluble in the epoxy resin before curing reactions start in order to obtain a homogeneous plastic. If a homogeneous blend is obtainable, then reaction rate characteristics become important, since the times required for gelation and hardening of the plastic must be within acceptable limits.

In addition to these basic requirements, numerous ancillary properties are desirable. The system components should not be so physiologically harmful that a manufacturing process using them would be hazardous, nor should the system be explosive or excessively flammable. Also, surface chemical and general physical properties of the system affect the quality of the finished item. The resin system should wet the reinforcing material well to achieve a strong bond, and it should have rheological properties appropriate to the filament winding process.

When a prospective curing agent is found to have usable physical and chemical properties, it is pertinent to consider any advantages that ensue from its use. That is, what mechanical characteristics does it impart to the composite structure which are improvements over competitive

materials? Previous reports in this series have considered this question in relation to some of the amine curing agents suitable for use in practical filament winding applications. Detailed analyses of the properties desired of a filament-winding matrix, based upon micromechanical measurements, are not presently available (9). However, in a great many applications a plastic matrix with a higher tensile modulus and ultimate tensile strength and, particularly, greater ductility at high yield levels would be superior to a matrix with lower values of these properties. Of course, other properties may be as significant, depending on the application of the filament-wound structure. Compression and impact properties, thermal and electrical properties, dynamic and fatigue properties, *etc.*, undoubtedly assume more importance in some applications.

The extent of possible use of a new curing agent ultimately depends on whether or not its advantages are worth the costs of making and using it. The costs may also determine whether it is used as a specialty item or one for general use.

#### AN EVALUATION OF *m*-AMINO BENZYL AMINE

MABA possesses enough of the desirable properties outlined in the above criteria to qualify for use in certain filament-winding applications. Since it is a low-melting solid, it is readily incorporated into epoxy resins of the Bisphenol-A or cycloaliphatic types. Once blended, a resin-MABA system does not precipitate nor otherwise become incompatible. MABA may also be blended with *m*-phenylene diamine to render a liquid two-component curing agent from which neither precipitates.

The molecular structure of MABA reveals that it is a "hybrid" amine, having one amino group that is aliphatic in nature while the other is aromatic. Its favorable properties probably result from this physicochemical dissymmetry.

Stoichiometric blends of MABA with the common epoxy liquids yield solutions with suitable processing viscosities. Reaction rates depend upon the type of epoxy employed. The blend with the diglycidyl ether of Bisphenol-A begins to react immediately—a characteristic of aliphatic amines. Successful filament winding with this system

usually requires provision for cooling the resin container. The reaction of MABA with cycloaliphatic epoxies is much slower, and no special cooling is required.

Very little is known about the toxicological properties of MABA, but since it is an aromatic amine, it is probably at least as toxic as aniline. Its volatility is quite low at ambient temperatures, and any vapor hazard posed by its use can be avoided by ventilation. It does not stain skin and clothing, as does *m*-phenylene diamine, but it probably can be absorbed through the skin. No instances of skin irritation or dermatitis have resulted from production and use of MABA, even though brief contact with the skin has occurred. Several aromatic amines are known to be carcinogenic, but the structural homologs of MABA are not believed to be dangerous in this respect.

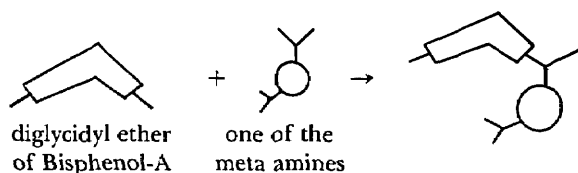
The particular merit of MABA is its ability to impart some desirable tensile properties to the most useful types of filament-winding plastics. Of the thermosetting plastics evaluated in this program, only that produced from MABA and the diglycidyl ether of Bisphenol-A exhibited a true tensile yield point. Other epoxy plastics of comparable modulus and ultimate tensile strength broke in a brittle manner. The plastic produced from MABA and the diepoxide of dicyclopentyl ether did not exhibit a yield point upon being elongated. However, its ultimate tensile strength is high, and the modulus is above the limits currently found even in fibrous synthetic plastics (10). The greater part of the increase, relative to the Bisphenol-A-type plastic, is attributable to the dicyclopentyl ether structure, but a significant portion of the increase is also contributed by MABA. This is illustrated by the comparison with the *m*-phenylene diamine cured system mentioned previously.

#### POSSIBLE REASONS FOR DUCTILITY DIFFERENCES

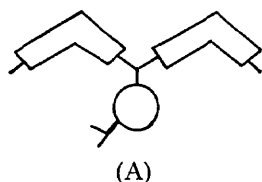
Since MXDA, MABA, and MPDA are all tetrafunctional reactants of similar structure, it is of interest to consider possible reasons for the greater ductility of the plastic produced from MABA and the diglycidyl ether of Bisphenol-A. Since this plastic exhibits a yield point, and its tensile modulus is slightly lower than that of the plastics produced from either MPDA or MXDA, it appears

that its molecular network is less rigid than those of the other plastics. This suggests that the crosslink density is reduced. A possible reason may be derived from a consideration of the reactions occurring in the early stages of gelation.

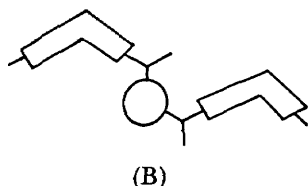
Expressed with symbols, the first reaction in any system is



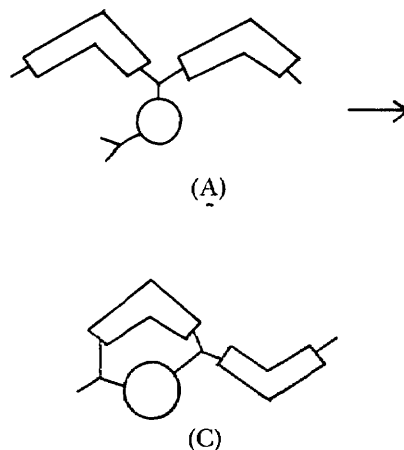
If the amine is MPDA or MXDA, this transient intermediate can only be a single entity, since the two amino groups are equivalent. However, with MABA, there are two possibilities, and it is probable that most of the initial reaction occurs at the aliphatic amino group. After the first step, the reaction obviously becomes quite complex, and there are several possible courses. Since epoxy molecules are in excess at this stage, a portion of a second intermediate should be



or



The steric and stoichiometric factors favor (B), particularly with MXDA or MPDA. With MABA, however, the reactivity of the aliphatic amino group should allow formation of a relatively large amount of the (A) form. Molecular models show that the (A) form has a very favorable geometry for intramolecular cyclization:



Although possible, steric conditions are not as favorable in the (B) form for this to occur.

This sequence of reactions must occur to some extent; even if only a small amount of (C) is formed, the crosslink density would be reduced, and bulky, linear segments would appear in place of chain intersections. A method of direct measurement of the disappearance of the amino hydrogen atoms of MABA and a synthesis of the cyclic structure are needed to substantiate or disprove this possibility. However, neither infrared nor nuclear-magnetic-resonance spectra of MABA differentiate the aliphatic and aromatic amino groups, and synthetic attempts have failed to yield a product which could be isolated and identified as the cyclic structure.

## CONCLUSIONS

Epoxy plastics based upon Bisphenol-A have limiting tensile strengths of about 12,000 psi and maximum elongations of about 7%. Some cycloaliphatic epoxies have limiting tensile strengths of about 18,000 psi and moduli of about 650,000 psi. The use of *m*-aminobenzyl amine as curing agent improves the elongation of the Bisphenol-A plastic without loss of tensile strength and with minor loss in modulus. The same curing agent improves the modulus and tensile strength of one isomer of cyclopentyl ether diepoxide without loss in elongation. This ability of a curing agent to improve the limiting properties of the two types of epoxy plastics of greatest importance is sufficiently unique to merit consideration.



The physical and chemical properties of *m*-aminobenzyl amine are such that it can be produced readily, preserved with a little care, and used reasonably easily in the production of filament windings and other composite structures.

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